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GAS-METAL REACTIONS IN ROCKET NOZZLES

Quarterly Progress Report
March 1 to June 1, 1962

Contract AF 33(616)-7744
Task No. 735101

Submitted to:

Wright Air Development Division
Wright-Patterson Air Force Base, Ohio

June, 1962

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GAS-METAL REACTION IN ROCKET NOZZLES

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Contract No. AF 33(616)-7744
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E. L. Olcott

Submitted To:
Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio

June, 1962

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ABSTRACT

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The continuing study of the reactions between refractory metal alloys and the combustion products of solid propellants is described. Four additional alloys and four additional propellants were selected. Tungsten-2 per cent thorium and tungsten-25 per cent rhenium alloy wires were used in both filament tests and optical bomb tests. The reactivity of tungsten-25 per cent rhenium in a CO/CO₂ mixture and in propellant combustion gases appeared to be less than other tungsten alloy. Nitriding reactions were found to be unimportant for tungsten, but tantalum and tantalum-10 per cent tungsten alloy showed considerable increases in hardness. Alternate procedures to test alloys not available as wire or thin sheet were considered and will be tried experimentally.

I. INTRODUCTION

This Quarterly Progress Report covers the first quarter of the extended program of study on the chemical reactions between solid propellant combustion gases and refractory metal alloys of interest for rocket motor nozzles. The scope of the program was broadened for this program extension to include a wider selection of refractory alloys and a greater variety of propellants typical of current and future formulations. During the first-year program, the study was limited to the reactions of tungsten, tantalum, and the tantalum-10 per cent tungsten alloy with the combustion products of three typical state-of-the-art propellants and one propellant containing a fluorocarbon binder.

Two laboratory tests were devised by which useful data on these reactions were collected. In the filament test, electrically heated refractory wires were exposed to individual gas species and simple mixtures. In the optical bomb test, refractory wires were exposed directly to the action of burning propellant strands under pressures similar to those found in rocket motors. Data on weight changes, physical changes, microstructural changes, and microhardness indices were used to describe the reactions which occurred.

In the current work, the same techniques are to be applied, insofar as possible, to a wider range of reaction systems. It is apparent that a number of refractory alloys of interest cannot be formed into small wires or thin sheets, and some modifications of the experimental methods will be required. Several techniques are available and will be applied to obtain meaningful reaction data.

II. SUMMARY

Two new alloy compositions, tungsten-2 per cent thorium and tungsten-25 per cent rhenium, were obtained in wire form. Two other alloys, tantalum-20 per cent tungsten and tungsten-15 per cent molybdenum, were obtained in bar form. Four new propellant formulations were selected for study to show the selective effect of certain combustion products and the effect of oxidative level of the combustion gases.

Filament tests were completed with the tungsten-2 per cent thorium and the tungsten-25 per cent rhenium in the CO-CO₂ system. The tungsten-25 per cent rhenium alloy appeared to be affected less by oxidation in a CO/CO₂ mixture than were other tungsten alloys. Exposure of tungsten, tantalum, and the tantalum-10 per cent tungsten alloy to nitrogen in filament tests did not produce any significant effect on tungsten but increased the hardness of tantalum and the alloy considerably.

The two new alloys available in wire form were also exposed to five different propellants (four propellants used last year and one new propellant formulation) in the Optical Bomb. The results were qualitatively similar among these tungsten alloys and similar to tungsten. The reactions of the tungsten-25 per cent rhenium alloy appeared to be less than for the unalloyed tungsten. Further analysis of the reaction products from these tests are planned.

Alternate experimental procedures were considered for use with alloy samples which cannot be obtained in wire or thin sheet form. These methods include (1) bar tests in rocket exhaust stream, (2) exposure of alloy chips to burning propellant in the Optical Bomb, and (3) plasma torch exposure of alloy rods with reactive gas injection. Experimental work with these techniques is planned to determine their usefulness.

III. ADDITIONAL ALLOY SELECTION

A number of refractory metal alloys, especially tungsten- and tantalum-based compositions, were considered for inclusion in this program. One immediate problem was apparent, however. Most alloys of interest are difficult to work, and many of them cannot be formed into fine wires or thin sheet. Since these are the only shapes directly applicable to the experimental techniques developed during the first year's work, the initial selection of alloys was limited. Two tungsten alloys which were found commercially available in wire or small rod form were tungsten-25 per cent rhenium and tungsten-2 per cent thoria. Experimental work was started with these two alloys using the existing procedures.

A number of organizations known to be working with refractory alloys were contacted in search of material samples. During the period of this report bar stock samples of tantalum-20 per cent tungsten (National Research Corporation) and tungsten-15 per cent molybdenum (Climax Molybdenum) were obtained. Personnel of the Metals Laboratory at Aeronautical Systems Division have agreed to provide samples of some alloys which are of interest in this study. Alternate experimental methods to be used with the bar stock samples are being considered and are discussed further in the last section of this report.

IV. ADDITIONAL PROPELLANT SELECTION

Additional propellants were selected for the current study to provide combustion gas compositions typical of several possible classes of developmental propellants. Propellant formulations were also chosen to show selectively the effects of the presence or absence of certain constituents in the combustion products. Thus, even if our estimates of future propellant technology are not entirely accurate, the data collected can give substantial guidance in the estimation of the effect of any propellant on the alloys studied.

Four propellant formulations selected for use in addition to those used and reported in the first year's work may be described as follows:

- (1) Chlorine-free Propellant - This formulation produces a chlorine-free, nitrogen-rich combustion gas, and is typical of those using oxidizers other than perchlorates. It also shows (by comparison) the role of HCl in corrosion of refractory alloys.
- (2) Aluminized Polyurethane Propellant A - This formulation is the first of a series of 6000°F flame temperature propellants selected to show the effect of propellant oxidation ratio. The oxidation level in this propellant is moderately high.
- (3) Aluminized Polyurethane Propellant B - Second member of the above series of 6000°F propellants. The oxidation level in this propellant is low (less oxidizing).
- (4) Very Fuel-Rich Propellant - This formulation produces free carbon in its combustion products, characteristic of one class of propellants with special fuel ingredients of possible future utility. The flame temperature of this propellant is low (about 4300°F), but the fuel-rich propellants it is intended to simulate generally have low flame temperatures, too (about 4500-4800°F).

The composition of the combustion gases from each of these propellants is shown in Table I. One or two other propellants may be added later in the program, if needed. Each propellant selected can be used to

TABLE I
GASEOUS COMBUSTION PRODUCTS OF ADDITIONAL PROPELLANTS

	Chlorine-free Propellant	Aluminized Polyurethane Propellants		Very Fuel-Rich Propellant
		A	B	
Flame Temperature	6080 °F	5975 °F	5975 °F	4280 °F
<u>Gas Species (Vol per cent)</u>				
H ₂	31.7	32.1	39.9	52.5
H ₂ O	2.9	11.7	4.9	0.018
CO	40.5	25.9	27.6	28.5
CO ₂	0.46	1.2	0.42	0.0015
HCl	0.00	11.5	8.4	0.75
HCN	--	--	--	0.38
N ₂	18.0	9.5	9.1	--
H	5.3	--	--	0.37
OH	0.27	0.91	0.33	--
Al	0.56	--	--	--
C ₂ H ₂	--	--	--	0.92
AlCl	--	--	--	14.7
C(Solid)	--	--	--	2.32 gm/100 gm propellant

expose wire samples in the Optical Bomb test, but fewer propellants may be used for the alternate methods of exposure needed for samples not available in wire form.

V. FILAMENT TESTS

Filament tests were conducted with the tungsten-2 per cent thorium and the tungsten-25 per cent rhenium wires in atmospheres containing CO_2 , CO, and a CO_2 -CO mixture. Each of these wires was also heated in argon to determine the effect of heat treatment alone. In these tests in argon some black volatile tungsten oxide generally forms because of the slow leakage of air into the test vessel. Each of the metals used in last year's work (tungsten, tantalum, and tantalum-10 per cent tungsten) were also heated in a nitrogen atmosphere to check for nitriding reactions. No significant effects of the exposure to nitrogen were observed in the case of tungsten. The tantalum and tantalum-10 per cent tungsten alloy showed considerable hardness increases up to 1250 KN*. Evidence of compound formation (presumably nitrides) was observed microscopically. The conditions and observations from each filament test are shown in Table II.

The most interesting observation apparent from the weight change data is the small effect of a CO/ CO_2 mixture (ratio of 31) on the tungsten-25 per cent rhenium alloy. A reduction in the weight loss of about 7-fold was noted between the CO_2 exposure and the CO/ CO_2 mixture exposure for this alloy. For the tungsten-2 per cent thorium alloy and for tungsten (reported last year), the reduction was only a little more than 2-fold. The tests with the tungsten-25 per cent rhenium were made at a lower filament temperature and should be repeated at a comparable temperature level to verify the effect.

Microscopic examination of the tungsten-rhenium wires after exposure to CO_2 mixtures showed a thin gray reaction layer. There is insufficient material for analysis, but it is likely an oxide of rhenium or a mixed rhenium-tungsten oxide.

* Knoop hardnesses were measured with a 25 gm load

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TABLE II
FILAMENT TEST DATA

Test No.	Material and Wire Size	Weight Difference (per cent)	Gas	Gas Pressure (mm Hg)	Temperature (°F)	Time (sec)	Observations (Macro)	Microscopic Observations Of Wire Cross-Section
A-83	25 mil tungsten	-0.55	N ₂	400	6000	300	Filament appeared etched. A small amt. of black deposit was found on tip of cold finger.	Thin crystalline layer on outside; no hardness change
B-57	25 mil tantalum	-0.07	N ₂	400	5000	300	No apparent effect on filament. Small amt. of black deposit on cold finger.	Small amount of dispersed phase. Hardness increase up to 1250 K; throughout.
C-48	24 mil tantalum-10% tungsten	-0.8	N ₂	400	5000	300	A metallic grey coating was on filament. Very slight amt. of white deposit was found on cold finger.	Same
D-3	20 mil tungsten-2% thorium	-39.8	CO ₂ Argon	20 380	5500	300	Large amt. of blue deposit on cold finger. Filament was greatly reduced in diameter.	Surface slightly pitted
D-4	20 mil tungsten-2% thorium	-17.6	CO ₂ CO	20 620	5500	300	Large amt. of dark blue deposit on cold finger. Filament was etched.	Surface slightly pitted
D-5	20 mil tungsten-2% thorium	-0.7	CO	400	5500	300	Slight amt. of black deposit on tip of cold finger. No apparent change in filament.	Surface slightly pitted
D-7	20 mil tungsten-2% thorium	-3.0	Argon	400	5500	300	Slight amt. of black deposit on tip of cold finger. Filament unaffected.	Surface slightly pitted
E-2	25 mil tungsten-25% rhenium	-26.6	CO ₂ Argon	20 380	4600	300	Large amount of blue deposit throughout chamber. Hard, black, crystalline, porous coating on filament.	Thin light grey reaction layer containing dispersed reflective particles.

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TABLE II
FILAMENT TEST DATA (Continued)

Test No.	Material and Wire Size	Weight Difference (per cent)	Gas	Gas Pressure (mm Hg)	Temperature (°F)	Time (sec)	Observations (Macro)	Microscopic Observations Of Wire Cross-Section
E-3	25 mil tungsten-25% rhenium	-3.8	CO ₂	20 620	4600	300	A blue deposit formed on the cold finger during the first minute. This was then covered with a black deposit. The filament was severely etched.	Thin light grey layer which penetrates the surface irregularly
E-4	25 mil tungsten-25% rhenium	-0.3	CO	400	4600	300	A small amount of black deposit formed on the cold finger. The filament was unaffected.	No surface change
F-5	25 mil tungsten-25% rhenium	-3.9	Argon	400	4600	300	Substantial amount of black deposit formed on the cold finger. Slight etching on ends of filament.	No surface change

VI. OPTICAL BOMB TESTS

Of the four new propellant formulations selected for this program, only one was used during this report period. Each of the two new alloy wires available to date (tungsten-2 per cent thorium and tungsten-25 per cent rhenium) were exposed to burning strands of the chlorine-free propellant (new) and the four propellants used in the program last year. Tungsten wires were exposed to strands of the five propellants taken from the same propellant batches for control purposes. The data from these tests are listed in Table III (tungsten), Table IV (tungsten-2 per cent thorium), and Table V (tungsten-25 per cent rhenium).

Many of the data and remarks based on macro-observations are similar for the alloys and for tungsten, with the notable exception of the effect of Arcite 368-A propellant on the tungsten-25 per cent rhenium. Here, as in the filament tests discussed above, the oxidation effect of this propellant seems substantially less for this alloy than for tungsten. This propellant, which is non-aluminized, produces a highly oxidizing combustion gas at a temperature of about 4700°F.

Microscopic examination of the wires exposed to the burning propellant in the Optical Bomb showed that in the case of Arcite 368 there was little reaction layer on any of the alloy wires. In Arcite 373 the tungsten and the tungsten-2 per cent thorium alloy showed similar behavior which was like that reported during the last period (formation of molten carbides which had high hardness and increasing hardness of the base metal at the interface). The tungsten-rhenium alloy was similar in its behavior except that the reaction layers were thinner and the over all weight change of the wire was less. No surface hardening effects could be measured with this alloy. The Arcocel 163 reaction layers were similar and like those reported in the last period. Again the tungsten-rhenium alloy showed somewhat less weight change. In the chlorine-free propellant all of the wire alloys behaved similarly in that they all showed a hard reaction

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TABLE III
DATA ON TUNGSTEN WIRES* EXPOSED
TO BURNING PROPELLANTS

Code	Propellant	Pressure (psi)	Weight Change (per cent)	Observations (Macro)	Microscopic Observation of Wire Cross-section
4A-5	Arcite 368A	300	-7.1	No apparent effect.	Thin grey reaction layer.
4A-6	Arcite 368A	1000	-4.5	No apparent effect.	Thin grey reaction layer.
5B-5	Arcite 373	300	+3.9	Four grey beads along wire.	Bead shows light grey and dark grey phases with hardness up to 2400 KN.
5B-6	Arcite 373	1000	+0.6	Glassy substance at both ends of wire.	Same as above
3C-5	Arcocel 163	300	-2.6	Some grey beads and one large lump on wire. Fused coating in one area.	Bead showed reflective layer with hardness of 1350 KN.
3C-6	Arcocel 163	1000	+11.0	One large lump and several grey beads along wire.	Bead showed reflective layer with hardness of 1325 KN. Several grey areas with the reaction layer.
2D-3	Chlorine-free	300	+3.9	One large grey bead and several small beads along wire.	Bead showed reaction layer with gray matrix and dispersed reflective particles. Reaction layer was 2000 KN hardness.
2D-6	Chlorine-free	1000	+7.7	Two grey beads and some glassy beads along wire. One area coated with a blue material.	Same as above.
2E-3	Fluorocarbon	300	+3.2	Some small yellow beads along wire.	Thin reaction layer containing reflective particles in a dark gray matrix.
2E-6	Fluorocarbon	1000	+3.8	Yellow beads and some blue coating along wire.	Thin reaction area containing grey and light grey particles.

* Wires were 25 mil diameter

TABLE IV
DATA ON TUNGSTEN-2 PER CENT THORIA WIRES
EXPOSED TO BURNING PROPELLANTS

Code	Propellant	Pressure (psi)	Weight Change (per cent)	Observations (Macro)	Microscopic Observations of Wire Cross-section
4A-2	Arcite 368A	1000	-3.9	No noticeable effect.	Thin crystalline outer layer.
5B-1	Arcite 373	300	+3.2	Grey beads along wire. Fused coating in two areas.	Bead shows light grey and dark grey phases with hardness up to 2400 KN.
5B-2	Arcite 373	1000	+3.2	A few small grey beads along wire. White glass-like beads and gold discoloration at one end.	Thin reaction layer contains a crystalline phase, a grey phase and reflective particle at the interface.
3C-1	Arcocel 163	300	-5.1	Two grey lumps and several grey beads along wire. Fused coating in one area.	Bead shows needlelike bright phase in grey matrix of high hardness.
3C-2	Arcocel 163	1000	+16.5	One large and some small grey beads along wire. Fused coat- ing in one area.	Bead shows grey reaction layer with faint needlelike pattern, 2000 KN hardness.
2D-1	Chlorine- free	300	-1.2	One large and some small grey beads along wire.	Bead showed grey reaction layer with dispersed reflective particles.
2D-4	Chlorine- free	1000	+30.7	Two small grey beads on wire.	Same as above. Hardness of reaction layer 2000 KN
2E-1	Fluorocarbon	300	+5.2	Many small yellow beads along wire.	Thin reaction layer containing reflective particles in a dark grey matrix. Thin crystalline layer on the outside.
2E-4	Fluorocarbon	1000	-23.7	Grey and brown beads along wire. Small portion of wire coated with a blue material.	Not examined.

* Wires were 20 mil diameter

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TABLE V.
DATA ON TUNGSTEN-25 PER CENT RHENIUM WIRES* EXPOSED
TO BURNING PROPELLANTS

Code	Propellant	Pressure (psi)	Weight Change (per cent)	Observations (Macro)	Microscopic Observation of Wire Cross-section
4A-3	Arcite 368A	300	0	No apparent effect.	Thin crystalline outer layer.
4A-4	Arcite 368A	1000	-2.6	No apparent effect.	Thin reaction layer-grey and light phases.
5B-3	Arcite 373	300	-0.1	Fused coating on one area.	Thin reaction layer-grey and light phases.
5B-4	Arcite 373	1000	+1.9	Glassy beads along wire. Fused coating in one area.	Thin reaction layer-grey and light phases.
3C-3	Arcocel 163	300	-3.9	Whole length of wire coated with fused coating and grey beads.	Bead showed reaction layer containing a grey phase, a light grey phase, and a dispersed reflective phase. Average hardness of layer is 1200 KN.
3C-4	Arcocel 163	1000	+5.1	Grey beads and black material over length of wire.	Bead showed reflective reaction layer of 1300 KN.
2D-2	Chlorine-free	300	+7.0	One large and one small grey bead on wire.	Bead shows reaction layer of a grey and reflective phase in a eutectic-like structure.
2D-5	Chlorine-free	1000	-21.8	One large grey lump and several small beads along wire.	Bead shows reflective particles in a grey matrix, 2000 KN hardness.
2E-2	Fluorocarbon	300	+3.8	One large yellow bead covered with grey slag on wire.	Bead showed a reaction layer containing light grey and dark grey particles.
2E-5	Fluorocarbon	1000	--	A few yellow beads and grey slag along wire.	Thin crystalline layer.

* Wires were 25 mil diameter

layer which consisted of a grey matrix with a dispersed reflective phase. Behavior in the fluorocarbon propellant was also similar among the three tungsten materials. Reaction layers were generally thin. Attempts will be made to analyze the yellow colored deposits found on all of the three tungsten materials after exposure to the fluorocarbon propellant.

VII. ALTERNATE EXPERIMENTAL TECHNIQUES

For alloy samples not available in wire or thin sheet form some modifications of the existing experimental methods will be needed. Two such alternate methods to expose samples to the action of hot propellant combustion products are under active consideration. One method is the use of bar specimens placed in the exhaust stream near the edge of the expansion cone in sub-scale solid propellant motor firings. This technique and the equipment needed for such exposures is available from another materials test program. Although this technique can be used, it does have some disadvantages. Relatively large specimens are needed for each test so the number of tests would necessarily be small. Also, since large quantities of propellant are required for a motor firing, the variety of propellants that could be used would be reduced. Thus, at this time, this technique is considered as a method to be used when other means cannot be found to test an alloy.

A second method of test under evaluation is the use of chips or turnings of an alloy mixed into a propellant composition before combustion of the propellant in the Optical Bomb. By recovering the chips from the bomb, metallographic examinations should be possible. The use of small tubes containing uncured propellant and the alloy chips will be tested as soon as a practical means of accomplishing this test method is devised.

The exposure of alloys to single gas constituents in the filament test, in other than wire form, does not appear practical. Data on those gas-metal combinations believed to be most important will be sought by using a plasma torch with secondary, reactive gas injection to heat a rod sample and expose it to reaction with the injected gas. The application of this technique may be limited by the need for relatively small and uniform test specimens to allow comparative results to be obtained. Empirical tests will be required to determine the utility of plasma torch tests.

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